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EVALUATION OF METALS DATA FOR SW-846 6010 (ICP-AES) ANALYSIS

1.0 SCOPE

l.l This procedure is applicable to inorganic data obtained from contractor laboratories analyzing metals by SW-846 Method 6010

l.2 The data validation is based upon analytical and quality assurance requirements specified in EPA SW-846, 3rd edition, Method 6010B.

2.0 RESPONSIBILITIES

Data reviewers will complete the following tasks as assigned by the Project Manager or Deputy Project Manager:

2.l. For a full (Tier 3) review :

2.l.l Data Assessment - "Total Review-Inorganics" Checklist Appendix (A.1). The reviewer must answer every question on the checklist.

2.l.2 Data Assessment - Data Assessment Narrative (Appendix A.2). The answer on the checklist must match the action in the narrative (appendix A.2) and on sample analysis result forms.

2.l.3 Data Review Log: It is recommended that each data reviewer maintain a log of the reviews completed to include:

a. date of start of SDG review
b. date of completion of SDG review
c. site name
d. SDG number
e. contract laboratory identifier
f. number of samples
g. matrix
h. hours worked
i. reviewer's initials

2.1.4 Telephone Record Log (telephone log) - the data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with contract laboratory. After the SDG review has been completed, attach a copy of the Telephone Record Log, along with copies of any subsequent associated laboratory submittals, to the completed Data Assessment Narrative (Appendix A.2).

3.0 DATA COMPLETENESS

Each data package is checked by the assigned data reviewer for completeness. A data package is considered to be complete when (a) all deliverables required per project scope are present, and (b) the contents of the electronic data deliverables match the hard-copy contents. If a data package is incomplete, the reviewer shall immediately notify the Project Manager for resolution. If the laboratory does not respond within 48 hours, the laboratory coordinator will be notified.

4.0 REJECTION OF DATA

All values determined to be unacceptable on the sample analysis result forms must be lined over with a red pencil. As soon as review criteria non-compliance causes data to be rejected, that data may be eliminated from any further review or consideration.

5.0 ACCEPTANCE CRITERIA

In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.l (pages 4-13) should be used.

APPENDIX A.1

		YES	NO	N/A
A.1.1	Cover Page - Present?	[___]	___	___
	Is cover page properly filled in, dated and signed by an *authorized signatory* of the laboratory?	[___]	___	

ACTION: If no, initiate telephone log, and contact laboratory for submittal.

A.1.2	Do sample numbers on cover page agree with sample numbers on:			
	(a) Analysis Request / Chain-of-Custody?	[___]	___	___
	(b) Sample Result Forms?	[___]	___	___

ACTION: If no for any of the above, contact laboratory for clarification / resolution.

A.1.3	Are all data summary forms labeled with:			
	Laboratory name?	[___]	___	___
	Sample and Laboratory ID Nos.?	[___]	___	___
	SDG No.?	[___]	___	___
	Correct units?	[___]	___	___
	Matrix?	[___]	___	___

ACTION: If no for any of the above, note omissions in the data assessment narrative.

A.1.4	Do any computation/transcription errors exceed 10% of reported values on sample result and QC reporting forms for: (NOTE: Check all forms against raw data.)			
	(a) all analytes analyzed by ICP?	[___]	___	___

ACTION: If yes, initiate telephone log, contact laboratory for corrected data and correct errors with red pencil and initial.

A.1.5	Raw Data			
A.1.5.1	Digestion Log* for ICP present? *Weights, dilutions and volumes used to obtain values.	[___]	___	___
	Are pH values present and < pH 2 for aqueous samples?	[___]	___	___
	Percent solids calculation present for soils/sediments?	[___]	___	___
	Are preparation dates present on sample preparation logs/bench sheets?	[___]	___	___

		YES	NO	N/A
A.1.5.2	ICP instrument read out record present?	[___]	___	___
A.1.5.3	Are all raw data to support all sample analyses and QC operations present?	[___]	___	___
	Legible?	[___]	___	___
	Properly Labeled?	[___]	___	___

ACTION: If no for any of the above questions in sections A.1.5.1 through A.1.5.3, initiate telephone log and contact laboratory for resubmittals.

A.1.6 Holding Times - (aqueous and soil samples)

(Examine sample traffic reports and analysis logs.)

ICP Metals analysis (6 months) exceeded ? ___ [___] ___

NOTE: Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of analysis (from raw data). Attach to checklist.

ACTION: If yes, reject (red-line) values less than Instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.

A.1.7 Is pH of any aqueous samples for:

Metals Analysis >2? ___ [___] ___

ACTION: If yes, flag the associated metals data as estimated.

A.1.8 Sample Results Forms

A.1.8.1	Are all sample results forms present and complete?	[___]	___	___

ACTION: If no, initiate telephone log and contact laboratory for submittal.

A.1.8.2	Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's?	[___]	___	___
	Are soil sample results for each parameter corrected for percent solids?	[___]	___	___
	Are all "less than IDL" values properly coded with "U"?	[___]	___	___

		YES	NO	N/A
	Are the correct concentration qualifiers used with final data?	[___]	___	___

ACTION: If no for any of the above, initiate telephone log, and contact laboratory for corrected data.

A.1.9	Are field sample ID #s and corresponding laboratory sample ID #s the same as on the Cover Page, sample results forms and in the raw data?	[___]	___	___
	Was a brief physical description of samples given on the sample results forms?	[___]	___	___
	Was the dilution factor of any sample re-analyzed at dilution noted on sample result form or on Run Log?	___	[___]	___

ACTION: If no for any of the above, note the omissions in the data assessment narrative.

A.1.10 Calibration

A.1.10.1	Is record of at least 2-point calibration present for ICP analysis (blank + at least one standard)?	[___]	___	___

ACTION: If no, initiate telephone log and contact laboratory for submittal.
If the 2-point calibration cannot be verified, Reject all associated results.

A.1.11 Initial and Continuing Calibration Verification

A.1.11.1	Present and complete for every metal?	[___]	___	___

ACTION: If no, initiate telephone log and contact laboratory for submittal.
If the ICV or CCV cannot be verified, Reject all associated results.

A.1.11.2	Circle on each ICV or CCV summary form all percent recoveries that are outside the acceptable limits of 90% to 110% of true value.			
	Are all calibration standards (initial and continuing) within control limits:			
	ICP metals: 90% - 110% recovery?	[___]	___	___

ACTION: Flag as estimated (J) all positive data (not flagged with a "U") analyzed between a calibration standard with %R between 75-89 recovery and nearest acceptable calibration standard. Qualify results <IDL as estimated (UJ) if the ICV or CCV %R is 75-89%. Reject (redline) as unacceptable data if recovery of the ICV or CCV is outside the range 75-125%. Qualify five samples on either side of verification standard out of control limits.

YES NO N/A

A.1.11.3 Was continuing calibration performed after daily initial calibration, after every 10 samples, and at the end of the analytical run? [___] ___ ___

ACTION: If no for any of the above, qualify the affected data, and note the problem in the "Data Assessment Narrative".

A.1.12 Initial and Continuing Calibration Blanks Summary Forms

A.1.12.1 Present and complete? [___] ___ ___

Was an initial calibration blank analyzed immediately following the daily ICV? [___] ___ ___

Was a continuing calibration blank analyzed after every 10 samples and at the end of the analytical run (following the final CCV)? [___] ___ ___

ACTION: If no, initiate telephone log, contact laboratory for submittal, and note the omission in the "Data Assessment Narrative".

A.1.12.2 Circle on each Blank Summary form all calibration blank positive values that are above 3x IDL or negative responses that are below 3x absolute IDL value.

ACTION: If no for any of the above, flag as estimated (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value above 3x IDL and nearest acceptable calibration blank.
Flag five samples on either side of the calibration blank outside the control limits.

A.1.13 Method (Digestion) Blank

A.1.13.1 Was one method blank analyzed for:

each Sample Delivery Group (SDG)? [___] ___ ___

each batch of digested samples? [___] ___ ___

each matrix type? [___] ___ ___

ACTION: If no for any of the above, flag as estimated (J) all the associated positive data for which prep. blank was not analyzed.

		YES	NO	N/A
A.1.13.2	Is concentration of any prep. blank value greater than any analyte PQL value?	___	[___]	___
	If yes, is the concentration of the sample with the least concentrated analyte less than 10x prep. blank value?	___	[___]	___

ACTION: If yes, reject (redline) all associated data greater than PQL but less than ten times the prep. blank value.

A.1.13.3	Is concentration of prep. blank value less than PQLs ?	[___]	___	___

ACTION: If no, reject (redline) all positive sample results when sample raw data are less than 10 times the prep. blank value.

A.1.13.4	Is concentration of prep. blank below negative PQLs?	___	[___]	___

ACTION: If yes, reject (redline) all associated sample results less than 10x IDL.

A.1.14 ICP Interference Check Sample Results

A.1.14.1	Present and complete?	[___]	___	___
	Was ICS analyzed at beginning of each run ?	[___]	___	___

ACTION: If no, flag as estimated (J) all the samples for which Al, Ca, Fe, or Mg is higher than the corresponding value in the ICS.

A.1.14.2	Circle all values on each ICS summary form that exceed $\pm$ 20% of true or established mean value.			
	Are all Interference Check Sample results inside the control limits ($\pm$ 20%)?	[___]	___	___
	If no, is concentration of Al, Ca, Fe, or Mg lower than the corresponding concentration in the ICS?	[___]	___	___

ACTION: (a) If any ICS recoveries are below 80%, qualify ('UJ' or 'J') all associated results; if ICS recoveries are significantly low (professional judgment), comment on the potential effects on the data in the data assessment narrative. (b) If any recoveries are above 120%, qualify ('J') all associated positive results; if ICS recoveries are significantly high (professional judgment), comment on the potential effects on the data in the data assessment narrative.

YES NO N/A

A.1.15 Matrix Spike Sample Results (Pre-Digestion)

A.1.15.1 Present and complete for: each SDG? [___] ___ ___

each matrix type? [___] ___ ___

ACTION: If no for any of the above, flag as estimated (J) all the positive data less than four times the spiking levels used for which spiked sample was not analyzed.

NOTE: If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J), if spike recoveries were acceptable (i.e., 75% - 125%).

A.1.15.2 Was field blank used for spiked sample? ___ [___] ___

ACTION: If yes, flag all positive data less than 4x spike added as estimated (J) for which field blank was used as spiked sample.

A.1.15.3 Circle on each Matrix Spike form all spike recoveries that are outside control limits (75% - 125%).

Are all recoveries within control limits? [___] ___ ___

If no, is sample concentration greater than or equal to four times spike concentration? [___] ___ ___

ACTION: If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on each Matrix Spike form for which sample concentration is less than four times the spike concentration.

Are results outside the control limits (75-125%) flagged by the laboratory on sample results and spike summary forms? [___] ___ ___

ACTION: If no, note the omissions in the data assessment narrative.

A.1.15.4 Are any spike recoveries:
(a) less than 75%? ___ [___] ___
(b) greater than125%? ___ [___] ___
(c) outside the documented historical acceptance limits for the particular matrix? ___ [___] ___

ACTION: (a) If any recoveries are below 75%, qualify ('UJ' or 'J') all associated results if the sample result is below 4x the spike concentration; if spike recoveries are significantly low (professional judgment), comment on the potential effects on the data in the data assessment narrative.

(b) If any recoveries are above 125%, qualify ('J') all associated positive results if the sample result is below 4x the spike concentration; if spike recoveries are extremely high, comment on the potential effects on the data in the data assessment narrative.
(c) If any recoveries are outside the documented historical acceptance limits, qualify the data appropriately and indicate potential bias strength and direction in the data assessment narrative.

A.1.16 Matrix Spike Duplicate Sample Results

A.1.16.1 Present and complete for: each SDG? [___] ___ ___

each matrix type? [___] ___ ___

ACTION: If no for any the above, flag as estimated (J) all positive results for which duplicate sample was not analyzed.

Note: (a) If one duplicate sample was analyzed for more than 20 samples, then first 20 samples do not have to be flagged as estimated, if duplicate precision values were acceptable.
(b) If percent solids for soil sample and its duplicate differ significantly (professional judgment), comment on the potential sample heterogeneity in the data assessment narrative.

A.1.16.2 Was field blank used for duplicate analysis? ___ [___] ___

ACTION: If yes, flag all positive data > 10x IDL as estimated (J) for which field blank was used as duplicate.

A.1.16.3 Are all values within control limits (maximum 20% RPD) or within the documented historical acceptance limits for each matrix? [___] ___ ___

Are results outside the control limits or outside documented historical acceptance limits flagged by the laboratory on sample results and duplicate summary forms? [___] ___ ___

ACTION: If no, note the omissions in the data assessment narrative.

NOTE: RPD is not calculable for an analyte of the spike – duplicate pair when both values are less than IDL.

A.1.16.4 Are any duplicate precision (%RPD) values:
(a) greater than 20%? ___ [___] ___
(b) outside the documented historical acceptance limits for the particular matrix? ___ [___] ___

ACTION: (a) If any %RPD are above 20%, qualify ('J') all associated positive results. If RPD values are extremely high (professional judgment), comment on the potential effects on the data in the data assessment narrative.
(b) If any recoveries are outside the documented historical acceptance limits, qualify the data appropriately and indicate potential effects on the data in the data assessment narrative.

A.1.17 Laboratory Control Sample (LCS)

A.1.17.1 Was an LCS prepared and analyzed for:

each SDG? [___] ___ ___

each batch samples digested? [___] ___ ___

each matrix type? [___] ___ ___

ACTION: If no for any of the above, initiate telephone log and contact laboratory for submittal of results of LCS. Flag as estimated (J) all reported results if LCS was not analyzed.

NOTE: If one duplicate sample was analyzed for more than 20 samples, then first 20 samples do not have to be flagged as estimated, if duplicate precision values were acceptable.

A.1.17.2 Are all values within the documented historical acceptance limits for each matrix? [___] ___ ___

A.1.17.3 Are results outside the documented historical acceptance limits flagged by the laboratory on sample results and duplicate summary forms? [___] ___ ___

NOTE: If IDL of an analyte is equal to or greater than true value of LCS, disregard the "Action" below even though LCS is out of control limits.

Is LCS "Found" value higher than the documented historical acceptance limits or greater than certified reference material acceptance limits? ___ [___] ___

ACTION: If yes, qualify all associated positive data as estimated (J).

Is LCS "Found" value lower than the documented historical acceptance limits or less than certified reference material acceptance limits? ___ [___] ___

ACTION: If yes, qualify all associated data as estimated (UJ or J).

YES NO N/A

A.1.18 Serial Dilution Samples

A.1.18.1 Was Serial Dilution analysis performed for:

each SDG? [___] ___ ___

each matrix type? [___] ___ ___

ACTION: If no for any of the above, flag as estimated all the positive data > 10x IDLs for which Serial Dilution Analysis was not performed.

A.1.18.2 Was field blank(s) used for Serial Dilution Analysis? ___ [___] ___

ACTION: If yes, flag all associated data > 10 x IDLs as estimated (J).

A.1.18.3 Are results outside control limit flagged on sample result forms and serial dilution summary forms when initial concentration is equal to 10 times IDL or greater ? [___] ___ ___

ACTION: If no, note the omissions in the data assessment narrative.

A.1.18.4 Circle on each serial dilution summary form all percent difference that are outside the control limits for initial concentrations equal to or greater than 10 x IDLs only.

Are any percent difference values > 10%? ___ [___] ___

ACTION: Flag as estimated (J) all the associated sample data > 10xIDLs for which percent difference is greater than 10%. If percent difference values are significantly higher than 10% (professional judgment), note the potential effects on the reported data in the data assessment narrative

Note: Flag on sample result forms only the sample results whose associated raw data are > 10xIDL

Note: As an alternate to the serial dilution, the method allows performance of a post-digestion spike addition for evaluation of potential chemical or physical interferences. If this alternate is used, the PSA recovery must be between 75% and 125% to verify the absence of interferences.

A.1.19 Verification of Instrumental Parameters

A.1.19.1 Are bi-annual (every six months) verification reports present for:

Instrument Detection Limits? [___] ___ ___

Evaluation of Metals Data for SW-846 6010 Analysis		YES	NO	N/A
ICP Interelement Correction Factors?		[___]	___	___
ICP Linear Ranges?		[___]	___	___

ACTION: If no, initiate telephone log and contact lab for submittal.

A.1.19.2 Instrument Detection Limits

A.1.19.2.1 Are IDLs present for:	all the analytes?	[___]	___	___
	all the instruments used?	[___]	___	___

ACTION: If no for any of the above, initiate telephone log and contact laboratory for submittal.

A.1.19.2.2 Is IDL greater than PQL for any analyte?	___	[___]	___
If yes, is the concentration on sample results form of the sample analyzed on the instrument whose IDL exceeds PQL, greater than 5 x PQL?	[___]	___	___

ACTION: If no, flag as estimated all values less than five times PQL of the instrument whose IDL exceeds PQL.

A.1.19.3 Linear Range Determinations

A.1.19.3.1 Was any sample result higher than high linear range of ICP.	___	[___]	___
Was any sample result higher than the highest calibration standard for non-ICP parameters?	___	[___]	___
If yes for any of the above, was the sample diluted to obtain the result on Form I?	[___]	___	___

ACTION: If no, flag the result reported on sample results form as estimated (J).

A.1.20 Percent Solids of Soils and Sediments

A.1.20.1 Are recalculated percent solids in soil or sediment samples within a reasonable error band, considering rounding error?	[___]	___	___

ACTION: If no, initiate telephone log and contact the laboratory for resolution.

APPENDIX A.2
DATA ASSESSMENT NARRATIVE

Project # _____ Site _____ Matrix: Soil _____
SDG# _____ Lab _____ Water_____
Contractor _____ Reviewer _____ Other_____

A.2.1 Validation Flags- The following flags have been applied in red by the data validator and must be considered by the data user.

J- This flag indicates the result qualified as estimated

Red- Line- A redline drawn through a sample result indicates unusable value. The redlined data are known to contain significant errors based on documented information and should not be used by the data user.

Unqualified Data- The results that do not carry "J" or "red-line" are unqualified for appropriate use.

A.2.2 The data assessment is given below and on the attached sheets.

A.2.2 (continuation)

APPENDIX A.3
INORGANIC DATA ASSESSMENT SUMMARY

SDG NO._____ SITE_____

LABORATORY_____ MATRIX_____

REVIEWER'S NAME _____ DATE _____

CHECKED BY _____ DATE _____

DATA ASSESSMENT SUMMARY

	ACCEPTABLE	QUALIFIED	REJECTED
1. HOLDING TIMES	_____	_____	_____
2. CALIBRATIONS	_____	_____	_____
3. BLANKS	_____	_____	_____
4. ICS	_____	_____	_____
5. LCS	_____	_____	_____
6. DUPLICATE	_____	_____	_____
7. MATRIX SPIKE	_____	_____	_____
8. SERIAL DILUTION	_____	_____	_____
9. SAMPLE VERIFICATION	_____	_____	_____
10. OVERALL ASSESSMENT	_____	_____	_____

ACTION ITEMS:_____

_____

_____

AREAS OF CONCERN:_____

_____

_____

1.0 PESTICIDE DATA VALIDATION REQUIREMENTS

This section presents data validation requirements for extractable pesticide compounds conducted using SW-846 Method 8081A (EPA 1996).

1.1 DATA PACKAGE COMPLETENESS AND CASE NARRATIVE

A case narrative should be included with each data package and should be reviewed for information specific to the reported data (e.g., missing or substituted documentation, nonconformances, abnormalities encountered with the samples, matrix problems, re-analyses, and deviations from the referenced analytical method).

1.2 INSTRUMENT CALIBRATIONS AND PERFORMANCE

The objective of initial and continuing calibration is to ensure that instrument conditions are adjusted properly to provide acceptable resolution, sensitivity, and accuracy for detecting target compounds prior to and throughout the analysis of samples. The GC must pass specific criteria prior to the analysis of samples to ensure maximum instrument sensitivity and chromatographic resolution specific to pesticide compounds.

1.2.1 Initial Calibrations (Levels D and E)

Initial calibration documents that instrument performance was acceptable prior to sample analysis. Initial calibration may be conducted by external or internal standardization methods. The information below provides guidance on the evaluation of both calibration methods.

Initial calibration criteria include the following:

- A five-point calibration curve must be established, with one concentration at or near the method DL.

- If the percent relative standard deviation (%RSD) is less than 20% over the working range, the average calibration factor can be used. If the %RSD over the working range exceeds 20%, a curve-fitting equation for calculating results must be employed. If curve-fitting is employed, the maximum %RSD shall be ~30%. If the laboratory used an average %RSD that was greater than 20%, qualify all associated positive sample results as estimated (J). For calculation of the calibration factor, see Appendix D.

- If the GC was calibrated using the internal standard technique, then at least one internal standard is analyzed in each calibration standard at the approximate concentration used for sample analysis and is free of matrix interference.

- Calibration standards are injected or purged in the same way as with the samples.

- All target compound peaks across the working range were integrated under the same conditions.

- If toxaphene, chlordane, BHC, or the DDT series were detected, review the calibration and quantitation information described in Section 7.6 of Method 8081A.

- Calibration standards are NIST-traceable (or equivalent).

Necessary documentation includes the following:

- Instrument identification, standard identification, calibration date, and standard analysis raw data

- Traceability certificates for all calibration standards (including a dilution log documenting the preparation), including standard identification, date of preparation, analyte, lot numbers, expiration date, and concentration values.

After evaluation is completed, qualify the sample results as follows:

- If the %RSD of the calibration factors for the initial calibration is >20% (>30% for curve fit calibrations), qualify the associated results for that compound as estimated (J, UJ).

- If the regression coefficients are less than 0.995 qualify any positive results for the associated compounds as estimated (J).

- If the minimum number of standards was not used for calibration (5 for average RRF and linear regression, 6 for second order polynomial, and 7 for third order polynomial) qualify all associated positive results as estimated (J).

- If the instrument was not calibrated before use, qualify all associated sample results as unusable (R, UR).

- If the raw calibration data are unavailable (i.e., cannot be provided by the laboratory) and continuing calibration data are either not available or are out of control, qualify the associated data as unusable (R, UR). If continuing calibration data is available and meets the requirements identified below, qualify all associated sample results as estimated (J, UJ).

- If traceability of calibration standards cannot be established or they were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

- If the calibration data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

1.2.2 Continuing Calibration (

Continuing calibration ensures that the instrument conditions are stable and that quantitative results are accurate.

Continuing calibration criteria are as follows:

- All standards were analyzed at the beginning of each analytical run, and a continuing calibration curve has been analyzed daily and after every 20 samples.
- Calibration or RF values or concentrations (regression quantitation models) are within 15%D of the initial calibration values.
- Continuing calibration compounds elute within the retention time windows of the initial calibration values.

Check standards are of known quality.

Necessary documentation includes the following:

- Instrument identification, standard identification, analysis date, and check standard analysis raw data
- For analyses using statistically determined acceptance criteria, derived control limit values
- Standard traceability certificates (including a dilution log documenting the preparation), including source identification, date of preparation, analyte, lot numbers, expiration date, and concentration values

After evaluation is completed, qualify all associated sample results as follows:

- If a continuing calibration check was not analyzed at the minimum frequency, qualify all associated sample results as estimated (J, UJ).
- If the calibration check RFs or concentrations are greater than +15%D of the initial calibration values and an acceptable calibration check has not been reanalyzed or the instrument has not been recalibrated, qualify all associated positive results as estimated (J). Non-detects require no qualification.
- If the calibration check RFs or concentrations are <75%D of the initial calibration values, and an acceptable calibration check has not been reanalyzed or the instrument has not been recalibrated, qualify all associated results as estimated (J, UJ).
- If the calibration checks do not fall within the retention time windows, associated sample results after the last in-control point may be affected. If no peaks are present within the retention time window of the deficient analyte of interest, no qualification is necessary. However, if peaks are present, qualify all affected sample results as unusable (R, UR).
- If the calibration check information is unavailable (i.e., cannot be provided by the laboratory), qualify all associated sample results as unusable (R, UR).

- If the calibration check data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

- If traceability of calibration standards cannot be established or they were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

1.2.3 Instrument Performance

Criteria for chromatographic resolution and instrument sensitivity are established to ensure the performance of the overall GC measurement system. These criteria are instrument-specific rather than sample-specific and should be met under all circumstances.

Instrument performance criteria include the following:

- At least three injections of all single-component standard mixtures and multi-response standards have been analyzed within a 72-hour period.

- The DDT and endrin breakdowns (or combined breakdowns) are 120% in all Evaluation B standard analyses.

After evaluation is completed, qualify the sample results as follows:

- If the DDT percent breakdown exceeds 20%, qualify all detected results for DDT as estimated (J) and all non-detects as unusable (UR) if DDD and DDE are detected. In addition, qualify all detected results for DDD or DDE as presumptive and estimated (NJ).

- If the endrin breakdown exceeds 20%, qualify all detected results for endrin as estimated (J) and all non-detects as unusable (UR) if endrin aldehyde or endrin ketone are detected. In addition, qualify all detected results for endrin ketone as presumptive and estimated (NJ).

1.3 BLANKS

Blank sample results are reviewed to assess the extent of contamination introduced through sampling, sample preparation, and analysis. Summarize all blank results in the validation narrative.

1.3.1 Calibration Blanks

Calibration blank results may not involve the same weights, volumes, or dilution factors as the associated samples because non-aqueous samples are reported in pg/kg units and the associated calibration blanks are reported in pg/L units. Therefore, it may be necessary to work from the raw data when reviewing the calibration blank data.

Blank analysis criteria are as follows:

- The calibration blank is performed immediately following a calibration check.

- Calibration blank results are <MDL.

- Calibration blank run after any sample where a target compound was present at levels that saturated the detector.

Necessary documentation includes instrument identification, calibration blank preparation/ analysis date, and calibration blank preparation/analysis raw data.

After evaluation is completed, qualify (applies only to results generated between the out-of-specification calibration blank and the nearest acceptable calibration blank) sample results as follows:

- If calibration blanks were not analyzed at the minimum frequency identified, qualify all associated sample results as estimated (J, UJ).

- If the absolute value of any negative calibration blank result exceeds the MDL, qualify all associated undetected sample results as estimated (UJ) and qualify associated positive sample results within two times the absolute blank value as estimated (J).

- If calibration blank results are >MDL but are less than the RL, qualify associated sample results as undetected (UJ) for any result >RDL but <5 times the highest blank concentration, qualify as estimated (J). Results >5 times the highest blank concentration do not require qualification.

- If the blank data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

1.3.2 Laboratory (Preparation) Blanks

Blank analysis criteria include the following:

- Laboratory blanks have been extracted and analyzed for each matrix and have been analyzed on each instrument at a minimum frequency of one per analytical batch. If cleanups are used, e.g. Fluorisil, sulfur cleanup is required, a method blank reflecting the cleanup process must also be analyzed for the batch in the same analytical batch (sulfur blank only if all samples in batch required sulfur cleanup).

- A laboratory blank was prepared at the same time as the samples using the same procedure, including any cleanup steps used.

- Laboratory blank results are <MDL.

Necessary documentation includes the following:

- Instzument identification and laboratory blank preparation/analysis raw data.

- Laboratory blank results, preparation and analysis dates, and MDL/RL values.

After evaluation is completed, qualify the sample results as follows:

- If a laboratory blank was not prepared with the associated samples at the minimum frequency identified, qualify all associated sample results as estimated (J, UJ).

- If calibration blank results are >MDL but are less than the RL, qualify associated sample results as undetected (UJ) for any result >RDL but <5 times the highest blank concentration, qualify as estimated (J). Results >5 times the highest blank concentration do not require qualification.

- If the blank data is incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

1.3.3 Field Blanks

Review the field sampling documentation to identify the field blank samples (usually referred to as equipment blanks) and sample types. If necessary, contact the project coordinator to obtain the required information. Verify that the field blanks were handled in the laboratory as actual samples. Positive results may indicate that decontamination procedures were inadequate or that contamination was inherent to the equipment used. No qualification is to be performed based on field blank results; however, the results should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision-making processes.

1.3.4 Trip Blanks

Review the field sampling documentation, if necessary, to identify the trip blanks. Review the report forms, quantitation reports, and chromatograms. Qualification of sample results is not required based on trip blank results; however, field blank results should be noted in the validation narrative to alert the data user to uncertainties in the data set during decision-making processes.

1.4 BIAS

Compliance with bias requirements is determined by laboratory performance and compliance with project-specific and analytical requirements, as determined by the analysis of MS/MSDs and surrogate compounds.

1.4.1 Surrogate Recovery

Surrogates provide a measure of performance on individual samples. Surrogate recovery criteria are as follows:

- Every sample that is analyzed is spiked with the appropriate surrogate compounds.

- Surrogate recoveries are within the specified laboratory limits or the limits of 50% to 150% if not specified.

- Surrogate materials are of known quality.

Necessary documentation includes the following:

- Surrogate results, preparation and analysis dates, and laboratory-established surrogate recovery limits

- Instrument identification and surrogate preparation/analysis raw data

- Final surrogate concentration, the amount of spike added and associated standard identification

- Traceability certificates (including a dilution log documenting the preparation), including identification, date of preparation, constituent, lot numbers, expiration date, and concentration values.

After evaluation is completed, qualify the sample results as follows:

- If surrogates were not added to the associated samples , qualify all associated sample results as estimated (J, UJ).

- Qualify all associated sample results as estimated (J, UJ) for surrogates out of specification but >l0% recovery. No qualification is required for non-detects associated with high recovery surrogates.

- Qualify all associated detected results as estimated (J) and non-detects as unusable (R) for surrogate recoveries <l0%, unless surrogates were diluted out (i.e., diluted below low-level ICAL standard levels) due to the presence of analyte concentrations requiring dilution to quantitate results. If surrogates are diluted out, qualify all associated results as estimated (J, UJ).

- If the surrogate data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

- If traceability of surrogate standards cannot be established or they were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

- If both method blank and sample surrogates are out of specification, note in the validation report narrative.

1.4.2 Matrix Spike Recovery

The MS/MSD results provide matrix-specific information on the accuracy of the method for specific target compound classes. The MS criteria are as follows:

- Matrix spikes were performed on a sample from each matrix present in the analytical batch at a minimum frequency of one per analytical batch.
- Matrix spikes were prepared at the same time as the associated samples in the same analytical batch, using the same procedures, including any cleanup steps used, and spike analytes were added as early in the sample preparation process as practicable.
- Matrix spike materials are NIST-traceable (or equivalent) whenever possible.
- Matrix spike percent recovery is within the laboratory-established limits or the limits of 50% to 150% if not specified.

Necessary documentation includes the following:

- Matrix spike results, preparation and analysis dates, and laboratory-established recovery limits
- Instrument identification and MS preparation/analysis raw data (Levels D and E only)
- Final matrix spike concentration, the amount of spike added, and associated standard identification
- Traceability certificates (including a dilution log documenting the preparation), including identification, date of preparation, constituent, lot numbers, expiration date, and concentration values.

After the evaluation is completed, qualify the sample results of similar matrix as the MWMSD samples according to Table 6-l.

Table 1-1. Pesticide MS/MSD Result Qualification. (2 Pages)

MS/MSD Recovery	Surrogate Recovery	Sample Result	Qualification
<LCL	Within limits	>5 times spike concentration	No qualification
		<5 times spike concentration and detected	J
		Undetected	UJ
<LCL	<LCL	<5 times spike concentration and detected	J

		Undetected	UJ
>UCL	<LCL	<5 times spike concentration limit and detected	U
		Undetected	UJ
>UCL	>UCL	<5 times spike concentration and detected	J
		Undetected	No qualification

- If a MS sample was not prepared with the associated samples at the minimum frequency identified, qualify all associated sample results as estimated (J, UJ).

- If the MS data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

- If traceability of MS standards cannot be established or they were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

- If the field blank has been used for spike analysis, note in the validation narrative.

- If it is determined from validation that only the spiked samples are affected by low recoveries (this may be obtained from the sample preparation sheet or the narrative), qualify only the results for the spiked sample as described above.

1.4.3 Laboratory Control Samples

A LCS or BSS serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation. Typically LCSs are used for non-aqueous sample matrices and should be similar to the matrix composition of the samples to be analyzed. Typically BSSs are used with aqueous samples and are spiked distilled waster.

The LCS/BSS criteria are as follows:

- A LCS/BSS was performed at a minimum frequency of one per analytical batch.

- A LCS/BSS was prepared at the same time as the associated samples in the same analytical batch, using the same procedures, including any cleanup steps used.

- The LCS/BSS standards are NIST-traceable (or equivalent) whenever possible. At a minimum, reagents used must be reagent grade or better.

- Results are within the published control limits or within the limits of 50% to 150% if not specified.

Necessary documentation includes the following:

- The LCS/BSS results and preparation and analysis dates
- Instrument identification and LCS/BSS preparation/analysis raw data
- Final concentration of the LCS/BSS, the amount of spike added to the LCS/BSS, and associated standard identification
- Traceability certificates (including a dilution log documenting the preparation), including identification, date of preparation, constituent, lot numbers, expiration date, and concentration values.

After evaluation is completed, qualify the sample results as follows:

- If the LCS/BSS recoveries are <LCL control limits, qualify all associated sample results as estimated (J for detects, UJ for non-detects).
- If the LCS/BSS recoveries are >UCL, qualify all associated positive sample results as estimated (J).
- If neither a LCS nor BSS sample was prepared with the associated samples at the minimum frequency identified, qualify all associated sample results as estimated (J, UJ).
- If the LCS/BSS data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).
- If traceability of LCS/BSS standards cannot be established or they were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

1.4.4 Performance Audit Samples

Performance audit samples are introduced to the laboratory as a normal field sample and are primarily used to evaluate the accuracy of the laboratory analytical procedure.

Contact the project coordinator for the identity, source, and control limits for any performance audit sample submitted with the sample group. Note the results of any performance audit sample in the validation narrative and summarize the results in the final data validation report.

1.5 PRECISION

Compliance with precision requirements is determined by the evaluation of MS and MSDs or a laboratory duplicate as described in the following subsections.

1.5.1 Matrix Spike/Matrix Spike Duplicate or Laboratory Duplicate Samples

Laboratory duplicate samples may consist of either a sample/replicate (i.e., the same sample prepared/analyzed twice in the analytical batch) pair or a MS/MSD (i.e., one sample spiked identically, prepared/analyzed in the analytical batch) pair. The frequency and logistics of laboratory replicates and matrix spike replicates are established in the program requirements. In the absence of this at least one precision evaluation sample should be included with every 20 field samples.

Duplicate (laboratory replicate or MS/MSD) sample criteria are as follows:

- The duplicate analysis was prepared at the same time as the associated samples in the same analytical batch, using the same procedures, including any cleanup steps used.

- The RPD must be <30% for aqueous samples (<50% for non-aqueous) for duplicate results >5 times the RL.

- For duplicate results <5 times the RDL, the range between the primary and duplicate results must be less than the RL for aqueous samples (<2 times the RL for non-aqueous).

Necessary documentation includes the following:

- Duplicate results and the preparation and analysis dates
- Instrument identification and preparation/analysis raw data.

After the evaluation is completed, qualify the sample as follows:

- If a duplicate sample was not analyzed with the samples at the minimum frequency identified, qualify all associated sample results as estimated (J, UJ).

- If the measured concentrations are both >5 times the RDL and the RPD is >20% for aqueous samples (>35% for non-aqueous), qualify all associated sample and duplicate results as estimated (J).

- If both sample and duplicate results are non-detects, no .qualification is required.

- If either or both of the measured concentrations are <5 times the RL, the above RPD criteria do not apply and the range between the sample and duplicate concentrations must be evaluated as follows:

    - If the range in concentration between the result(s) or reporting limit(s) is < RL value for aqueous samples (2 times the RL value for non-aqueous matrices), no qualification is required.

- If the range in concentration between the result(s) or reporting limit(s) is >RL value for aqueous samples (2 times the RL value for non-aqueous samples), qualify associated sample results as estimated (J) for detects. Non-detects are not qualified.

- If field blanks were used for laboratory duplicates, note in the validation narrative.

- If the duplicate data are incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

1.5.2 Field Duplicate Samples

The collection of field duplicate (collocated) samples are specified for some sampling events. If a field duplicate sample is sent to the laboratory, the results can aid in the overall evaluation of the data set. The validator shall be provided with the identification of collocated samples or shall obtain identification information where this process is required in the program.

The default RPD limits for the field duplicates (where both results are >5 times the RL) are 30% for water samples and 50% for soils. When one or both the results are <5 times the RL, the default limit should be expressed as the difference between result and RL value or the difference between the RL values, in which the acceptable limits are the range of RDL for water samples and 2 times RL for soils. Data qualification is not required for field duplicate RPD; however, the results of field duplicates should be discussed in the validation narrative to alert data users to uncertainties in the data set during decision-making processes.

1.5.3 Field Split Sample

A field split sample is used primarily to assess precision. A field split sample is a representative sample from a sampling event sent to a third-party (reference) laboratory. If so required by the program, the validator shall contact the project coordinator for the identification of the field duplicate submitted to the laboratory if the information has not already been provided.

The reference laboratory data are used to help formally evaluate the project data quality objectives at the end of the data validation process and are not specifically used to qualify an individual data package. Evaluate the field split sample results by comparing the corresponding sample results to the reference laboratory sample results. Note the results of the split sample duplicate analysis in the validation narrative, and summarize the results in the final data validation report.

1.6 SYSTEM PERFORMANCE

During sample analysis and between instrument performance and internal QC checks, conditions in the measurement system can affect the usability of sample data. Therefore, a review of additional data quality indicators must be performed to identify problems that may affect the interpretation and usability of the subject data. Evaluate system performance by reviewing the following types of information:

- Review the report forms, chromatograms and quantitation reports for evidence of GC/EC baseline anomalies, retention time shifts, extraneous peaks, low resolution, and peak anomalies.

- Check that positive results are not affected by abrupt changes in baseline caused by leaks in the injector system or GC column bleed.

If in the validator's professional judgment quantitative sample results may be biased due to system performance anomalies, such judgment must be addressed in the validation narrative and the affected results shall be qualified accordingly.

1.7 HOLDING TIMES AND SAMPLE PRESERVATION

The analyte-specific holding time and sample preservation criteria are shown in Table 6-2. For any analyte not included in this table, contact the project coordinator for specific criteria.

Table 1-2. Pesticide Analytes, Method Identification, Holding Times,

Analytical Parameters	Method	Holding Time[a]	Preservation
Gas Chromatography			
Pesticides	8081A[b]	Aqueous 7/40[c] Nonaqueous 14/40	All – Cool to 4°C
TCLP Pesticides	1311/8081	Aqueous 7/40 Nonaqueous 14/7/40[d]	All – Cool to 4°C

a. Holding time in days (unless otherwise noted).
b. Four-digit numbers from Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (EPA 1986).
c. First time to initial sample extraction, second time from extraction to analysis.
d. First time to TCLP extraction, second time from TCLP extraction to preparation extraction, and third time from preparation extraction to analysis.

Necessary documentation includes the sampling date and preservation (normally on sample chain-of-custody) and preparation/analysis dates.

After evaluation is completed, qualify results as follows:

- If samples were not preserved and were not analyzed within identified holding times, qualify all affected sample results as unusable (R, UR).

- If samples were not preserved and were analyzed within identified holding times, qualify all affected sample results as estimated (J, UJ).

- If properly preserved samples analyzed past the identified holding times but <2 times past the identified holding times, qualify all affected sample results as estimated (J, UJ).

- If properly preserved samples were analyzed >2 times past the identified holding times qualify all affected detected sample results as estimated (J) and non-detected results as unusable (UR).

1.8 SAMPLE RESULT COMPOUND IDENTIFICATION, QUANTITATION, AND DETECTION LIMITS

Qualitative criteria have been established to minimize false positives and negatives in the reporting of pesticide data. These criteria include compliance with dual, dissimilar column quantitation, retention time window criteria on dissimilar columns and GC/MS confirmation if the sample concentration for any single pesticide is at least 10 parts per million (ppm) in the sample extract.

1.8.1 Compound Identification and Quantitation

Compound identification and quantitation criteria are as follows:

- All positive results are within the retention time windows.
- Positive results were analyzed and reported on dissimilar columns.
- If interference is evident, the lower of the two values are reported.
- If no interference is evident, the higher of the two values are reported.
- The pattern for multi-peak pesticides (e.g., chlordane and toxaphene) matches the standard chromatograms.
- Results are within the linear range of the instrument calibration.
- Proper extraction techniques were used for aqueous and non-aqueous samples and for TCLP samples. Jar extraction was conducted for TCLP, and that the proper extraction fluid was used based on the preliminary evaluation of the waste sample.
- If samples were analyzed using the internal standard technique, internal standard recovery limits are established by the laboratory, or use 50% to 150%.

Required documentation includes the following:

- Laboratory sample results, preparation/analysis dates, and DL values for non-detected analytes
- Instrument identification and sample preparation/analysis raw data (Levels D and E only).

After evaluation is completed, qualify the sample results as follows:

If the qualitative criteria are not met, qualify detected results as non-detect as follows: if the misidentified peak is outside the retention time windows and no interferences are noted, report the RL; if the misidentified peak interferes with a target peak, then the reported value is qualified as estimated and undetected (UJ).

If detected results have not been analyzed on dissimilar columns, qualify the results as unusable (R).

- If quantitation and confirmation are questionable, all affected data should be qualified as presumptive and estimated (NJ).

- If GC/MS confirmation was required but not conducted, note this fact in the validation reports and also note the effect on the sample results.

- Check calculations and correct any sample results as necessary.

- If results are reported from analyses that are outside of the linear calibration range of the instrument, qualify results as estimated (J, UJ).

- If samples were analyzed using the internal standard technique and the recoveries exceed limits, qualify the associated data as estimated (J, UJ). If internal standard recovery limits are not provided by the laboratory, note as such in the validation narrative.

- If sample preparation cannot be verified or if sample preparation was conducted improperly and no other major or minor deficiencies are identified, qualify the associated results as estimated (J, UJ) and note as such in the data validation package and final report (Levels D and E only).

- If the validator determines that incorrect identifications were made as a result of cross-contamination or carryover between analyses, then the affected data should be qualified as unusable (R, UR) and noted as such in the validation narrative.

1.8.2 Reported Detection Limits

Result RL value criterion includes the reported MDL values meeting the RL or client-specific requirements.

Required documentation may include any or all of the following items: preparation/analysis dates, MDL study information, MDL values, and RL values for non-detected analytes.

After evaluation is completed, qualify results as follows:

- Note in the validation report which RL values for non-detects do not meet the method or client-specific values.

- If sample results and RL values cannot be verified, qualify all affected results as estimated (J, UJ).

- If systematic errors are discovered, request clarification from the project coordinator and note the results in the validation reports.

1.9 SAMPLE CLEANUP

Sample cleanup procedures are used to remove matrix interferences. Gel permeation chromatography is frequently used to remove high molecular weight interferents. FluorisilB is frequently used to remove polar compound interferents. Other solid phase absorbants (e.g., alumina, silica gel) used for sample cleanup must meet the criteria defined for Fluorisil except for the criteria that Fluorisil check solutions contain 2,4,5-trichlorophenol.

Sample cleanup criteria includes the following:

- Fluorisil cartridges or bulk material have every lot number checked before use.

- All anlaytes of interest in the check solutions.

- Analyte of interest recoveries 80- 110%.

- Fluorisil check solutions contain 2,4,5-trichlorophenol.

- 2,4,5-tichlorophenol recoveries < 5%.

- GPC columns checked before use.

- All anlaytes of interest in GPC check solutions.

- GPC analyte of interest recoveries should be 80-l 10%.

- GPC column calibrated before use.

- GPC column calibration checked once every 7 days or before use.

- Check materials used are of known quality.

- All associated analytical batch QC samples (e.g., blanks, matrix spikes, LCS/BSS) also received the same cleanup as the samples

Necessary documentation includes the following:

- Lot or batch numbers, check/calibration material identification, analysis dates, check/calibration material analysis results and raw data.

- Analytical batch cleanup logs/raw data

- Check/calibration material traceability certificates including a dilution log documenting the preparation including source identification, date of preparation, analyte, lot numbers, expiration date, and concentration values.

After the evaluation is completed, qualify the samples results as follows:

- If the initial check did not meet the specified recovery, qualify all associated sample results as estimated (J, UJ). If recovery is 0 qualify all associated non-detected results as rejected (UR)

- If the initial check information is unavailable (cannot be provided by the laboratory), qualify all associated sample results as unusable (R, UR).

- If the initial check data is incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

- If a GPC calibration check was not analyzed at the minimum frequency, qualify all associated sample results as estimated (J, UJ).

- If the GPC calibration check internal standards do not fall within the retention time windows, qualify all affected detected sample results as estimated (J) and rejected for non-detects (UR)

- If the GPC calibration check data is unavailable (cannot be provided by the laboratory), qualify all associated sample results as unusable (R, UR).

- If the GPC calibration check data is incomplete or calculations cannot be confirmed, qualify all associated sample results as estimated (J, UJ).

- If traceability of check/calibration materials cannot be established or were used past their expiration date, qualify all associated sample results as estimated (J, UJ).

- If the associated analytical batch QC samples were not given the same cleanup, qualify all associated sample results as estimated (J, UJ).

1.10 OVERALL ASSESSMENT AND SUMMARY

Complete the data validation checklist (Appendix A) and summarize the data validation results according to the requirements of Section 10.0.

PESTICIDE DATA VALIDATION CHECKLIST

Validation Tier	II	III			
Project:			Data Package:		
Validator:		Lab:		Date:	
Case:			SDG:		
Analyses Performed					
SW-846 8081	SW-846 8081 (TCLP)	SW-846 8081 (TCLP)			
Samples/Matrix					

1. Data Package Completeness and Case Narrative

Technical verification documentation present?	Yes	No	N/A

Comments:

2. Instrument Performance and Calibrations

Initial calibration acceptable?	Yes	No	N/A
Continuing calibrations acceptable?	Yes	No	N/A
Standards traceable?	Yes	No	N/A
Standards expired?	Yes	No	N/A
Calculation check aqcceptable?	Yes	No	N/A
DDT and endrin breakdowns acceptable	Yes	No	N/A

Comments:

3. Blanks

Calibration blanks analyzed?	Yes	No	N/A
Calibration blank results acceptable?	Yes	No	N/A
Laboratory blanks analyzed?	Yes	No	N/A
Laboratory blank results acceptable?	Yes	No	N/A
Field/trip blanks analyzed?	Yes	No	N/A
Field/trip blank results acceptable?	Yes	No	N/A

Transcription/calculation errors?	Yes	No	N/A

Comments:

4. Bias

Surrogates analyzed?	Yes	No	N/A
Surrogate recoveries acceptable?	Yes	No	N/A
Surrogates traceable?	Yes	No	N/A
Surrogates expired?	Yes	No	N/A
MS/MSD samples analyzed?	Yes	No	N/A
MS/MSD results acceptable?	Yes	No	N/A
MS/MSD standards NIST traceable?	Yes	No	N/A
MS/MSD standards expired?	Yes	No	N/A
LCS/BSS samples analyzed?	Yes	No	N/A
LCS/BSS results acceptable?	Yes	No	N/A
Standards traceable?	Yes	No	N/A
Standards expired?	Yes	No	N/A
Transcription/calculation errors?	Yes	No	N/A
Performance audit sample(s) analyzed?	Yes	No	N/A
Performance audit sample results acceptable?	Yes	No	N/A

5. Precision

Duplicate RPD values acceptable?	Yes	No	N/A
Duplicate results acceptable?	Yes	No	N/A
MS/MSD standards NIST traceable?	Yes	No	N/A
MS/MSD standards expired?	Yes	No	N/A
Field duplicate RPD values acceptable?	Yes	No	N/A
Field split RPD values acceptable?	Yes	No	N/A
Transcription/calculation errors?	Yes	No	N/A

Comments:

6. System Performance

Chromatographic performance acceptable?	Yes	No	N/A

Positive Results resolved acceptable?	Yes	No	N/A

Comments:

7. Holding Times

Samples properly preserved?	Yes	No	N/A
Sample holding times acceptable?	Yes	No	N/A

Comments:

8. Compound Identification, Quantitation, and Detection Limits

Compound identification acceptable?	Yes	No	N/A
Compound quantitation acceptable?	Yes	No	N/A
Results reported for all requested analyses?	Yes	No	N/A
Results supported in the raw data?	Yes	No	N/A
Samples properly prepared?	Yes	No	N/A
Detection limits meet RDL?	Yes	No	N/A
Transcription/calculation errors?	Yes	No	N/A

Comments:

9. Sample Cleanup

Fluorisil® (or other absorbant) cleanup performed?	Yes	No	N/A
Lot check performed?	Yes	No	N/A
Check recoveries acceptable?	Yes	No	N/A
GPC cleanup performed?	Yes	No	N/A
GPC check performed?	Yes	No	N/A
GPC check recoveries acceptable?	Yes	No	N/A
GPC calibration performed?	Yes	No	N/A
GPC calibration check performed	Yes	No	N/A
GPC calibration check retention times acceptable?	Yes	No	N/A
Check/calibration materials traceable?	Yes	No	N/A
Check/calibration materials expired?	Yes	No	N/A
Analytical batch QC given similar cleanup?	Yes	No	N/A
Transcription/calculation errors?	Yes	No	N/A

Comments:

Comments:

Table . DQO Summary Table

STEP 1	STEP 2	STEP 3	STEP 4	STEP 5	STEP 6	STEP 7
State the Problem	Identify the Decisions	Identify the Inputs to the Decisions	Define Study Boundaries	Develop Decision Rules	Specify Tolerable Limits on Errors	Optimize Sampling Design